

The Determination of Trace Metals in Engine Oil Using the GTA-95 Graphite Tube Atomizer

Application Note

Atomic Absorption

Author

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Introduction

The importance of the determination of trace metals in oil samples has been widely recognized by the Petroleum Industry [1]. However the analysis of oil samples using atomic absorption presents problems not encountered with aqueous samples [2].

In most cases, to prevent lengthy digestion procedures, the sample is dissolved in an organic solvent and either aspirated into the flame or dispensed directly onto a graphite furnace atomizer. With flame atomic absorption the diluting solvent can act as a fuel and markedly disturb the flame conditions. As a result long aspiration times may be required for steady state conditions to be reached.

Electrothermal atomization provides a number of advantages over flame methods and these include:

1. The use of a wide range of diluting or extracting solvents
2. Microlitre sample consumption
3. Greater sensitivity
4. Complete destruction of the sample matrix
5. Automatic standard preparation.



Agilent Technologies

Scope

This article covers the parameters and conditions required for the determination of Al, Cr, Cu, Ni, Pb and V in engine oil after dilution of the oil sample in diisobutylketone (DISK). Emphasis is placed upon the determination of volatile elements such as lead (Pb) which can be lost with the oil matrix during the ash phase. This loss can be overcome by the use of a Pyrolytic Graphite Platform inserted into the furnace. The platform permits the direct determination of lead in oil without a lengthy acid digestion [3].

Instrumental requirements for the flame atomic absorption and sample preparation procedures for oil samples dissolved in organic solvents are presented in Varian Instruments at Work AA-10 [4]. Further information on the analysis of petroleum products for trace metals using furnace techniques can be obtained by consulting "Analysis of Petroleum and Petroleum Products by Atomic Absorption Spectroscopy and Related Techniques" [1] and "Analysis of Petroleum for Trace Metals" [5].

Instrumentation

Spectrophotometers	Agilent AA-975, AA-075
Graphite furnace	Agilent GTA-95 and Programmable Sample Dispenser
Desk top computer	HP-85 and Graphics Tape
Printer	HP-82905A
Recorder	Agilent 9176

Standard Preparation

Different instrumental responses occur with different organometallic compounds. Therefore the accuracy of the analysis can depend on the chemical form of the standards [1]. Conostan organometallic standards are prepared from alkylarylsulfonates in a base oil and are available as individual concentrates or multi-element blends. Conostan standards are extremely stable, soluble in ketones, paraffinic and aromatic hydrocarbons, easy to handle and have been used for the calibration of instruments in spectrometric methods. Suppliers of oil soluble standards are listed in the references [6,7]. When preparing standards and samples in organic matrices the following suggestions and practical hints should be considered.

1. The standards should be made up in accordance with the manufacturer's instructions [6,7].
2. The standards can be prepared on a weight per weight, weight per volume, or volume per volume basis in the

chosen organic solvent. It is therefore necessary to quote the method and solvent used as the concentration units are not equivalent. Conversion to other concentration units can be easily determined if the density of the diluting organic solvent is known.

3. Many of the standards prepared in oil are extremely viscous and difficult to handle in the preparation of stock solutions. The oil may have to be heated for use with an autodiluter. A convenient method employed in our laboratory is to weigh out accurately by difference the required amount of oil soluble standard using a 5 mL plastic syringe and make up with either the required volume or mass of the organic solvent.
4. The physical nature of the standards and the samples must be matched. This can be achieved by adding the required amount of blank oil to the standards.
5. The analytical zero is established against a blank made up from unused oil in accordance with the sample preparation procedure.
6. Ideally, the standards and sample should give a response between 0.1 and 0.8 Abs. This can normally be achieved by appropriate dilution and/or by selecting a wavelength for the element that corresponds with the response and required concentration range.
7. In this study all samples and standards were dispensed using an autosampler. The rinse solution for the autosampler can be the diluent solvent or a mixed solvent capable of rinsing the dispensing capillary system. Alternatively, water containing an appropriate surfactant (Triton X100 0.01% w/v) has also been used effectively in this study as a rinse solution [3].

Atomization from Tube Wall

Each oil sample will have a characteristic background signal depending on the analytical wavelength, the ashing conditions and the nature of the sample. In this study the engine oil sample (1 g oil in 100 mL Diisobutylketone (DIBK)) gave a great deal of smoke during the ashing stage and the resulting background signal was found to be independent of wavelength. Shown in Figure 1 is the relation between the background signal and ashing temperature for the dissolved engine oil sample. The maximum permissible ashing temperature before the loss of analyte for each element under investigation is also indicated.

Figure 1 shows that a temperature greater than 600 °C is required to ensure adequate ashing of the oil sample when the sample is dispensed directly onto the tube wall. At this temperature Pb is lost with the ashing products.

This does not permit the determination of lead in the oil sample when dispensed onto the tube wall directly. However this loss of lead can be overcome by using the pyrolytic graphite platform inserted in the graphite furnace. The conditions for the determination of lead are presented in Part A.

The appropriate ashing conditions for Cu, Ni, Al, Cr and V can easily be selected provided these metals are not present as volatile metallo-organic species such as porphyrins. In cases where porphyrins are present the ash temperature for each element has to be investigated and set accordingly. The conditions for the determination of these elements directly from the tube wall are presented in Part B.

Chromium in Engine Oil
AA-875 GTA-95
1g Oil in 100ml DIBK
357.9nm

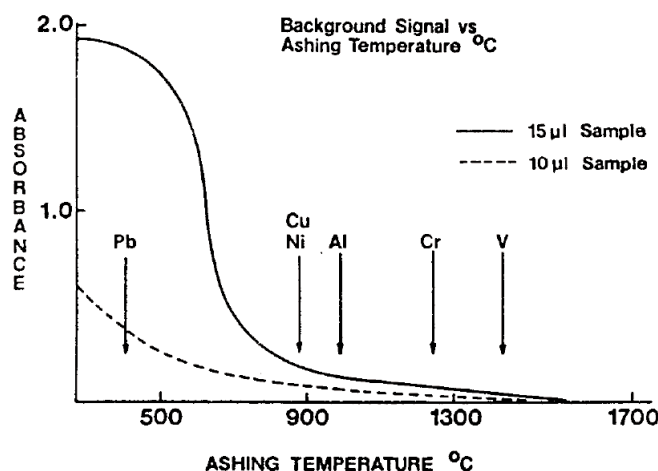


Figure 1. Background signal vs ashing temperature. Maximum ashing temperature of various elements together with the background signals generated by 15 μ L and 10 μ L of diluted oil sample atomized from a pyrolytic coated graphite tube.

It is also interesting to note that an increase in the sample volume by 50% in going from 10 μ L to 15 μ L causes an increase in background signal of about 150%. The effect of sample volume on the background signal should be investigated with any new sample.

For best precision, reproducible drying and minimum background signal it is recommended that between 5 and 10 μ L be used in most analyses for samples directly injected onto the tube wall. For greater analytical response the multiple dry and ash facility on the GTA-95 autosampler can be used.

Part A

Atomization of Lead from Pyrolytic Graphite Platform

The determination of lead can be achieved without further sample treatment by using a pyrolytic graphite platform which can be easily inserted into the Graphite Tube (Figure 2). The platform has been shown to reduce many chemical and background interferences experienced with volatile elements [3].

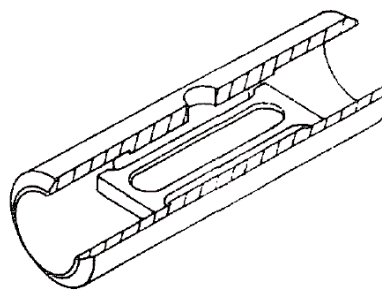


Figure 2. Pyrolytic graphite platform inserted into a graphite tube.

Investigation of a 5 μ L sample (1 g engine oil in 100 mL DIBK) atomized from the tube wall with a set ash temperature of 350 $^{\circ}$ C showed a background signal of about 0.4 Absorbance concurrent with an atomic lead peak (Figure 3). The secondary wavelength (283.3 nm) was used in preference to the 217.0 nm line as it provides improved signal to noise and reduces background absorption.

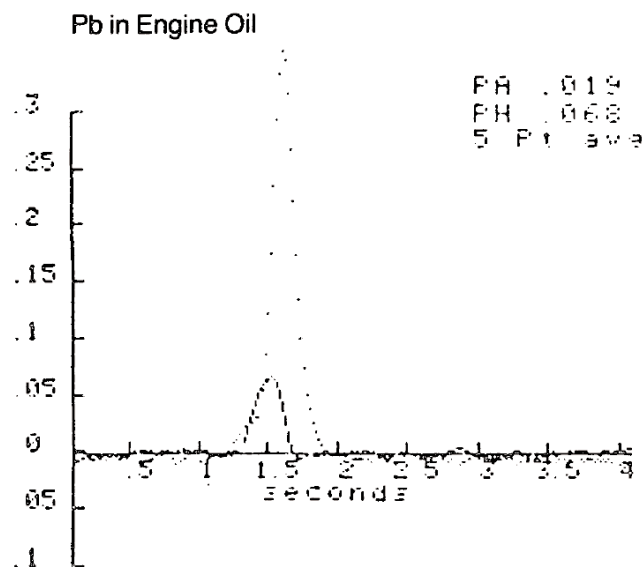


Figure 3. 5 μ L of 1 g engine oil in 100 mL DIBK atomized from the tube wall.

When the same sample was atomized from the pyrolytic platform the response improved 2.5 times and the background signal fell to zero (Figure 4). This suggests that with atomization from the tube wall some lead was lost, either occluded in ashing products of the sample or as a molecular species.

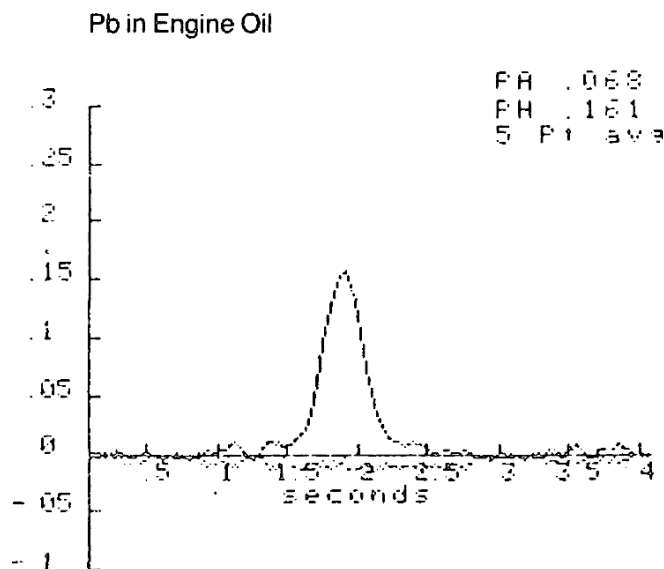


Figure 4. 5 μ L of 1 g Engine Oil in 100 mL DIBK atomized from the pyrolytic graphite platform.

Further investigation showed that the platform could reproducibly accommodate up to 40 μ L of diluted sample. With a 30 μ L sample no background signal was found (Figure 5). This indicates that it is possible to dissolve a higher amount of oil in the diluent solvent when the pyrolytic graphite platform is used, permitting a lower level of determination of lead in the original sample.

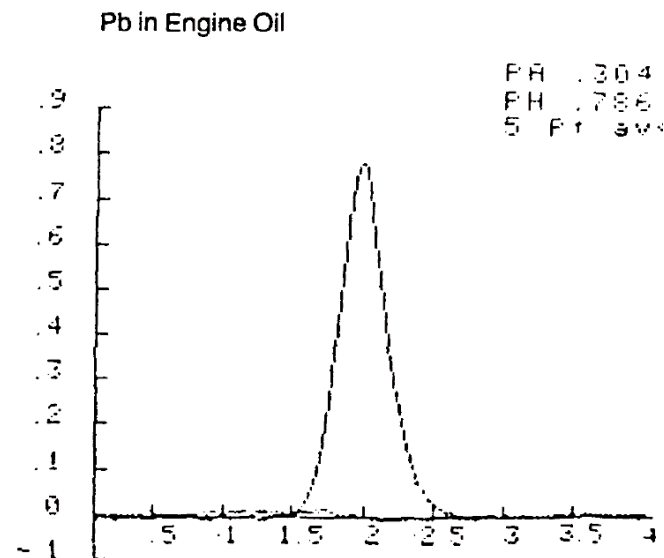


Figure 5. 30 μ L of 1 g Engine Oil in 100 mL DIBK atomized from the pyrolytic graphite platform.

The temperature program for the pyrolytic platform was different from that employed when atomizing the sample directly from the tube wall [2]. The platform lags the tube wall temperature and, as a result, the programmed tube temperatures have to be set higher for most steps when the platform is used (Table 1). Pb would be lost above 350 $^{\circ}$ C with atomization from the tube wall, yet with the platform an ash temperature of 500 $^{\circ}$ C (steps 3 to 5) was found to be optimum. The atomize temperature (steps 6 to 7) was 2400 $^{\circ}$ C with the platform, while from the tube wall 2000 $^{\circ}$ C was used. A cool down step (step 9) was inserted to allow the platform to reach ambient temperature before the next sample was dispensed.

Table 1. Program on The GTA-95 for the Determination of Pb in Engine Oil Using the Pyrolytic Platform

FURNACE OPERATING PARAMETERS.

STEP NO.	TEMPERATURE $^{\circ}$ C.	TIME SEC.	GAS FLOW	GAS TYPE	READ COMMAND
1	90	3.0	3.0	NORMAL	
2	160	40	3.0	NORMAL	
3	500	10	3.0	NORMAL	
4	500	5.0	3.0	NORMAL	
5	500	1.0	.0	NORMAL	
6	2400	1.0	.0	NORMAL	*
7	2400	1.0	.0	NORMAL	*
8	2400	2.0	.0	NORMAL	*
9	40	11	3.0	NORMAL	
10					

Calibration for the Determination of Lead

In order to gauge the effect of the oil matrix on the determination of lead, a standard addition method (Table 2) was compared with a normal calibration method (Table 3).

Table 2. GTA-95 Autosampler Conditions for the Standard Addition Determination of Lead in 1 g Engine Oil Dissolved in 100 mL DIBK Using a 100 ng/mL Pb Organometallic Standard

FURNACE OPERATING PARAMETERS.

STEP NO.	TEMPERATURE $^{\circ}$ C.	TIME SEC.	GAS FLOW	GAS TYPE	READ COMMAND
1	90	3.0	3.0	NORMAL	
2	160	40	3.0	NORMAL	
3	500	10	3.0	NORMAL	
4	500	5.0	3.0	NORMAL	
5	500	1.0	.0	NORMAL	
6	2400	1.0	.0	NORMAL	*
7	2400	1.0	.0	NORMAL	*
8	2400	2.0	.0	NORMAL	*
9	40	11	3.0	NORMAL	
10					

The linear range for the standard addition method was up to 0.5 Abs. The sample and addition volumes were selected to accommodate this range. The autosampler permitted the direct preparation of the standard addition samples from a stock standard and the sample. In this analysis an organometallic stock standard solution of 100 ng/mL Pb in DIBK was used [7]. 4 μ L, 8 μ L and 12 μ L of this standard was added to a 5 μ L volume of sample and made up to 17 μ L with blank to give ADDN 1, 2 and 3 respectively. Addition zero, the sample, was made up of 5 μ L of sample and 12 μ L of blank. Making up the addition to a constant volume with the blank ensures a valid analytical zero and corrects for possible contamination by the diluent solvent.

Table 3. TA-95 Autosampler Conditions for the Normal Calibration Using a 40 ng/mL Pb Organometallic

FURNACE OPERATING PARAMETERS.

STEP NO.	TEMPERATURE °C.	TIME SEC.	GAS FLOW	GAS TYPE	READ COMMAND
1	90	3.0	3.0	NORMAL	
2	160	40	3.0	NORMAL	
3	500	10	3.0	NORMAL	
4	500	5.0	3.0	NORMAL	
5	500	1.0	.0	NORMAL	
6	2400	1.0	.0	NORMAL	*
7	2400	1.0	.0	NORMAL	*
8	2400	2.0	.0	NORMAL	*
9	40	11	3.0	NORMAL	
10					

The autosampler was also used to prepare the normal calibration set of standards from an organometallic stock solution. A series of standards ranging from 0 ng/mL to 40 ng/mL Pb was prepared from a stock solution of 40 ng/mL Pb and blank prepared in DIBK.

The calibration graphs obtained from the programs presented in Tables 2 and 3 are presented in Figure 6. These two calibration graphs should be parallel if there is no interference from the oil sample. In this case the graphs are parallel within the precision of the method indicating negligible interference from the sample matrix.

Summaries of the analysis by both methods for Pb in the same oil sample using different standard stock solutions are presented in Tables 4 and 5. By standard additions the level of lead in the sample was calculated to be 9.2 μ g Pb/g while by normal calibration 9.3 μ g Pb/g. This clearly shows that for the determination of Pb in this oil sample the normal calibration method could have been employed because the matrix has negligible influence.

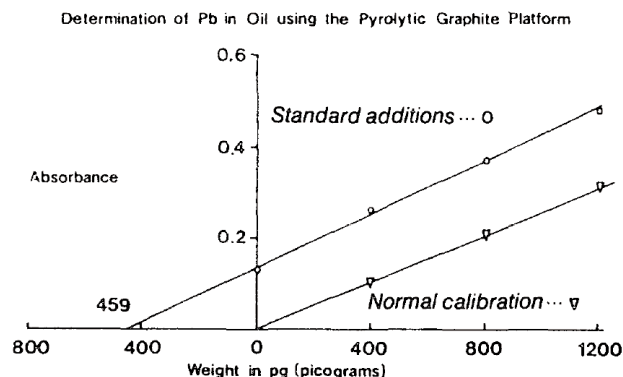


Figure 6. Comparison of standard additions and normal calibration Methods for programs listed in Tables 2 and 3.

Table 4. Standard Additions Calibration for Pb on Pyrolytic Platform

PYROLYTIC GRAPHITE PLATFORM

Pb in Engine Oil
(1 g Oil in Diisobutylketone)
STANDARD ADDITION
Addition Solution 100 ng/mL Pb
Average of 3 replicates
ADDN 1 400 pg 0.260 Abs
ADDN 2 800 pg 0.374 Abs
ADDN 3 1200 pg 0.485 Abs
ADDN 0 0 pg 0.130 Abs
Regression Line
 $A = MC + As$ $M = 0.000295 \text{ Abs/pg}$
 $As = 0.135 \text{ Abs}$
Wt Pb in 5 μ L diluted sample = 459 pg
Pb in oil sample = 9.2 μ g/g

Table 5. Normal Calibration for Pb on Pyrolytic Platform (Figure 6)

PYROLYTIC GRAPHITE PLATFORM

Pb in Engine Oil
(1 g Oil in Diisobutylketone)
STANDARD ADDITION
Addition Solution 100 ng/mL Pb
Average of 3 replicates
ADDN 1 400 pg 0.260 Abs
ADDN 2 800 pg 0.374 Abs
ADDN 3 1200 pg 0.485 Abs
ADDN 0 0 pg 0.130 Abs
Regression Line
 $A = MC + As$ $M = 0.000295 \text{ Abs/pg}$
 $As = 0.135 \text{ Abs}$
Wt Pb in 5 μ L diluted sample = 459 pg
Pb in oil sample = 9.2 μ g/g

Part B

Atomization of Cu, Ni, Al, Cr and V from the Tube Wall

The sample volume and nature of the sample can play an important role in determining the ashing conditions when the sample is dispensed directly onto the tube (Figure 1). The atomic and background signals for chromium were well resolved for both 15 μL and 10 μL volumes of the diluted oil sample over the temperature range 300 $^{\circ}\text{C}$ – 1700 $^{\circ}\text{C}$. The ashing study for chromium (Figure 7) shows that when a low ashing temperature was used (300 $^{\circ}\text{C}$ – 700 $^{\circ}\text{C}$) with a 15 μL volume, loss of atomic chromium occurred. Chromium is lost on atomization, either as a molecular or occluded species, with the residual matrix. This phenomenon has also been observed for nickel in oil and more recently in the determination of aluminium in serum [10]. It is therefore necessary to examine the effect of ash temperature on the atomic signal to ensure there is no loss of the analyte. For a 10 μL sample Figure 7 indicates that an ash temperature greater than 300 $^{\circ}\text{C}$ but less than 1200 $^{\circ}\text{C}$ is appropriate while for a 15 μL sample the ash temperature has to be set between 700 $^{\circ}\text{C}$ and 1200 $^{\circ}\text{C}$.

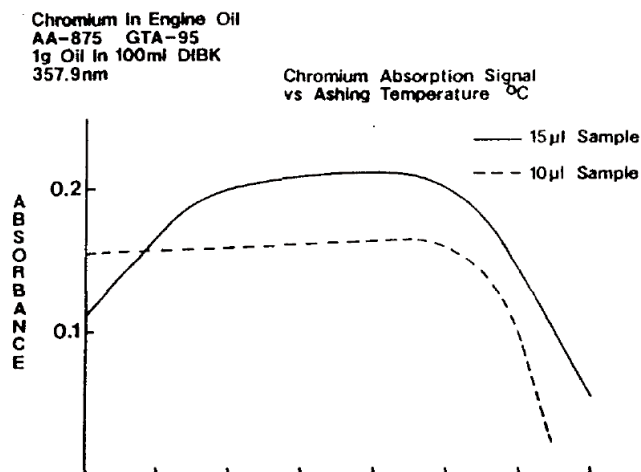


Figure 7. Atomic signal response for Chromium for 1 g Engine Oil dissolved in 100 mL DIBK at various ashing temperatures atomized from the tube wall.

Listed in Table 6 are the furnace parameters for the analysis of Cr in engine oil directly dispensed on to the graphite tube. Steps 1–4 are the dry phase and were selected by using a mirror to observe drying of the sample in the graphite tube, and adjusting the furnace parameters to ensure that an even drying of the sample occurred. Steps 5–7 represent the ashing phase, and steps 8–9 the atomize stage. Gas stop was used to maximize residence time and peak absorbance of the analyte in the graphite tube. Step 10 was a tube cleaning step.

Table 6. Furnace Parameters for the Determination of Chromium in Engine Oil Dissolved in 100 mL DIBK Using a 20 Ng/mL Cr Organometallic Standard

FURNACE OPERATING PARAMETERS.

STEP NO.	TEMPERATURE $^{\circ}\text{C}$	TIME SEC.	GAS FLOW	GAS TYPE	READ COMMAND
1	40	10	3.0	NORMAL	
2	85	5.0	3.0	NORMAL	
3	98	20	3.0	NORMAL	
4	265	15	3.0	NORMAL	
5	700	5.0	3.0	NORMAL	
6	700	10	3.0	NORMAL	
7	700	1.0	.0	NORMAL	*
8	2600	1.0	.0	NORMAL	*
9	2600	2.0	.0	NORMAL	*
10	2600	2.0	3.0	NORMAL	

SAMPLER PARAMETERS

NORMAL CALIBRATION

SAMPLES AND STANDARDS TYPE	LOCATION	VOLUME	BLANK VOLUME	MODIFIER VOLUME
BLANK	--	--	10	
STD 1	46	10		
STD 2	47	10		
STD 3				
STD 4				
STD 5				
SAMPLES	--	10		

LAST SAMPLE NO. = 5 MULTIPLE INJECTIONS = 1
NO. OF REPLICATES = 3 LAST DRY PHASE STEP = 2
RESLOPE RATE = INJECTION TEMP. =AMB
SINGLE SAMPLE NO. = 1

For the determination of Cu and Al, only the atomize and tube clean steps (8–10) were changed (Table 7).

In the analysis of nickel and vanadium in the oil sample, Conostan metallo-organic standards were used. Nickel and vanadium were not detected in the diluted sample; however, it was possible to determine furnace parameters from a series of oil matched standards. Table 7 summarizes the GTA-95 conditions required for each element when 1 g of oil is dissolved in 100 mL of DIBK.

Table 7. Summary of GTA-95 Furnace Conditions for a 10 μL Sample of 1 g Engine Oil Dissolved in 100 mL DIBK Atomized from the Tube Wall. Background Correction Was Used Together with Standard Operating Conditions [2,9]

Element	Ash $^{\circ}\text{C}$	Atomize $^{\circ}\text{C}$	Sensitivity (pg)
Cr	700	2600	2
Al	700	2500	7
Cu	700	2300	5
Ni	900	2400	13
V	1100	3000	29

Ramp Rate 2000 $^{\circ}\text{C}/\text{s}$, Gas stop during atomize

Figures 8–12 show the results of the determination of Cr, Al and Cu in engine oil. National Spectrographic Laboratories standards were diluted to give the appropriate concentrations and blank oil added to each standard to give the same concentration of oil as the diluted sample. The concentrations listed in Figures 8–12 are for the diluted samples in ng/ml and are levels above the blank. The level of analyte in the original oil sample is 100 times the value determined in the diluted sample.

00	CH	-0.013	C
00	CH	-0.012	C
00	CH	-0.011	C
MEAN	CH	-0.012	C
SDEV	CH	0.001	C
ZERO	CH	0.000	C

01	CH	0.101	C
01	CH	0.175	C
01	CH	0.172	C
MEAN	CH	0.176	C
SDEV	CH	0.005	C
STD 1	CH	10.	C

02	CH	20.	C
02	CH	19.	C
02	CH	20.	C
MEAN	CH	19.	C
SDEV	CH	1.	C
STD 2	CH	20.	C

01	CH	7.	C
01	CH	8.	C
01	CH	7.	C
MEAN	CH	8.	C
SDEV	CH	0.	C

02	CH	5.	C
02	CH	6.	C
02	CH	5.	C
MEAN	CH	5.	C
SDEV	CH	0.	C

03	CH	9.	C
03	CH	9.	C
03	CH	9.	C
MEAN	CH	9.	C
SDEV	CH	0.	C

04	CH	4.	C
04	CH	4.	C
04	CH	4.	C
MEAN	CH	4.	C
SDEV	CH	0.	C

OVER	CH	25.	C
OVER	CH	25.	C
OVER	CH	25.	C
MEAN	CH	25.	C
SDEV	CH	0.	C

Chromium in Engine Oil

AA-875 GTA-95

Wavelength

357.9 nm

Slit

0.5 nm

Lamp Current

5.0 mA

CONC

ng/mL

BC ON

PEAK HEIGHT

Sample (1 g oil in 100 mL DIBK)

Sample volume 10 µL

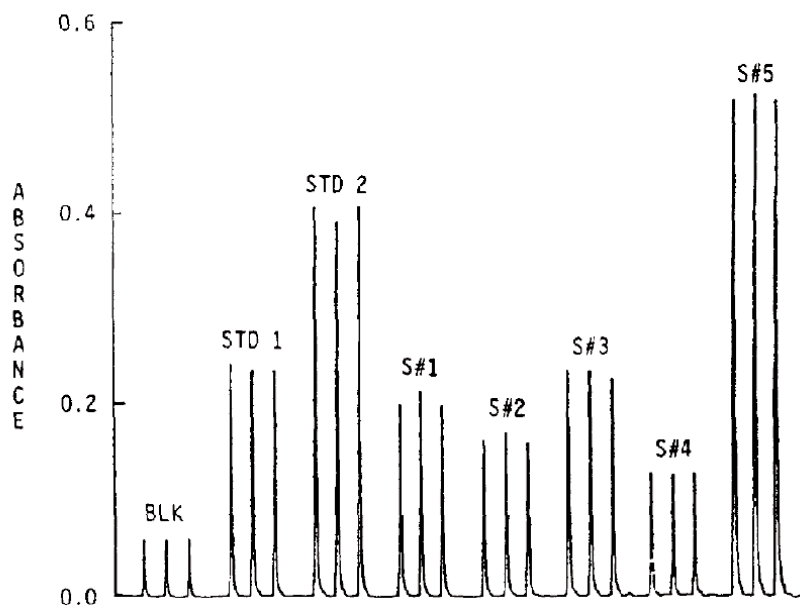


Figure 8. Traces for the determination of chromium in engine oil (Table 6).

00	CH	0.010	C
00	CH	-0.004	C
00	CH	-0.011	C
MEAN	CH	-0.002	C
SDEV	CH	0.011	C
ZERO	CH	0.000	C

01	CH	0.285	C
01	CH	0.277	C
01	CH	0.279	C
MEAN	CH	0.280	C
SDEV	CH	0.004	C
STD 1	CH	50.	C

02	CH	79.	C
02	CH	78.	C
02	CH	80.	C
MEAN	CH	79.	C
SDEV	CH	1.	C
STD 2	CH	100.	C

01	CH	37.	C
01	CH	36.	C
01	CH	37.	C
MEAN	CH	37.	C
SDEV	CH	0.	C

02	CH	13.	C
02	CH	12.	C
02	CH	13.	C
MEAN	CH	13.	C
SDEV	CH	1.	C

Aluminium In Engine Oil

AA-875 GTA-95

Wavelength

309.3 nm

Slit

1.0 nm

Lamp Current

7.0 mA

CONC

ng/mL

BC ON

PEAK HEIGHT

Sample (1 g oil in 100 mL DIBK)

Sample volume 10 μ L

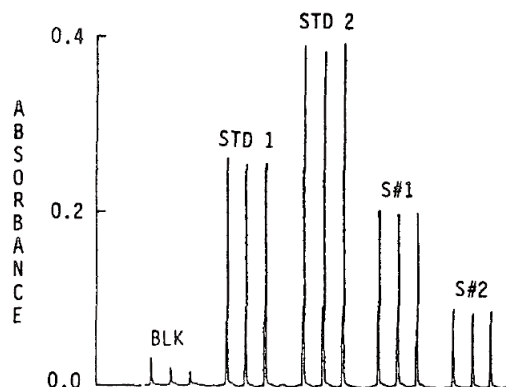


Figure 9. Traces for the determination of aluminium in engine oil.

Copper In Engine Oil

AA-875 GTA-95

Wavelength

324.8 nm

Slit

0.5 nm

Lamp Current

3.5 mA

CONC

ng/mL

BC ON

PEAK HEIGHT

Sample (1 g oil in 100 mL DIBK)

Sample volume 10 μ L

00	CH	-0.033	C
00	CH	-0.029	C
00	CH	-0.025	C
MEAN	CH	-0.029	C
SDEV	CH	0.004	C
ZERO	CH	0.000	C

01	CH	0.316	C
01	CH	0.316	C
01	CH	0.306	C
MEAN	CH	0.313	C
SDEV	CH	0.006	C
STD 1	CH	50.	C

02	CH	92.	C
02	CH	94.	C
02	CH	93.	C
MEAN	CH	93.	C
SDEV	CH	1.	C
STD 2	CH	100.	C

01	CH	15.	C
01	CH	15.	C
01	CH	15.	C
MEAN	CH	15.	C
SDEV	CH	0.	C

02	CH	21.	C
02	CH	22.	C
02	CH	21.	C
MEAN	CH	21.	C
SDEV	CH	0.	C

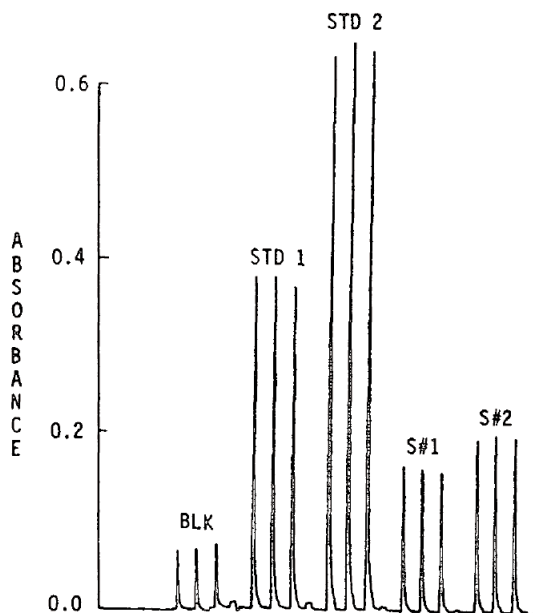


Figure 10. Traces for the determination of copper in engine oil.

Conclusion

Lead and other volatile elements can be lost during the ash phase of an oil sample dispensed directly onto the graphite tube. This loss can be eliminated by using the pyrolytic graphite platform, permitting the normal calibration determination of volatile elements. The less volatile elements such as Al, Cr, Cu, Ni and V can be determined in engine oil by dispensing the sample directly onto the wall of the graphite tube.

Acknowledgment

I would like to thank Dean Johnson for presenting this paper at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy in 1983 [11].

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